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MEMORANDUM

DATE: 18 January 1999

TO: David Bennett, WAM, U S EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle
Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data
Laboratory Batch. K9805842
Site. Duwamish River

WORK ASSIGNMENT NO: 46-35-0JZZ

WORK ORDER NO : 4000-019-038-5200-00

DOC. CONTROL NO : 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U S EPA, Region X
Dena Hughes, Site Manager, WESTON, Seattle (memo only)
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of nine sediment samples, laboratory batch K9805842, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered.

98354060	98354066	98354067	98354070	98354072
98354074	98354083	98354085	98354086	

Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240 1, February 1994), modified to include specific requirements of analytical methods

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QA Review Batch K9805842 (Organotin)

Site Duwamish River

Page 2

1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples were extracted within 13 days after sample collection, exceeding the 7 day holding time criteria in the Sampling and Analysis Plan. However, prior to extraction, samples were stored frozen, thus extending the holding time. Samples were extracted within the 12 month holding time recommended by PSEP for frozen samples. All samples met extraction holding time criteria but some samples exceeded analysis holding time criteria. Samples were extracted and analyzed as follows:

Sample ID	Date Collected	Date Extracted	Date Analyzed	No. of Days
98354060	8/26/98	9/2/98	9/14/98	7 days (extraction) 12 days (analysis)
98354066	8/27/98	9/2/98	9/14/98	6 days (extraction) 12 days (analysis)
98354067	8/27/98	9/2/98	9/14/98	6 days (extraction) 12 days (analysis)
98354070	8/27/98	9/9/98	9/16/98	13 days (extraction) 7 days (analysis)
98354072	8/27/98	9/9/98	9/16/98	13 days (extraction) 7 days (analysis)
98354074	8/27/98	9/9/98	9/17/98	13 days (extraction) 8 days (analysis)
98354083	8/27/98	9/2/98	9/14/98	6 days (extraction) 12 days (analysis)
98354085	8/27/98	9/2/98	9/14/98	6 days (extraction) 12 days (analysis)
98354086	8/27/98	9/2/98	9/14/98	6 days (extraction) 12 days (analysis)

Sample results and detection limits were qualified as estimated (U/J) when analysis holding times were exceeded.

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QA Review Batch K9805842 (Organotin)

Site. Duwamish River

Page 3

2. Detection Limits

Detection limits met project required quantitation limits with the following exceptions.

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98354060	n-Butyltin	10	60

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference.

3. Initial Calibration

A seven-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD

4 Continuing Calibrations

Continuing calibration check was performed after every 10 samples. Target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent (75-125 percent recovery) with the following exceptions:

Sample	Compound	% Recovery	Associated Samples
CCV2	Dibutyltin	131	98354074

As the associated sample was reanalyzed under acceptable conditions, no qualifiers were assigned based on continuing calibration results

5 Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks

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QA Review Batch K9805842 (Organotin)

Site: Duwamish River

Page 4

b) Field Blanks

No field blanks were associated with this SDG.

6. Surrogate Compound Recovery

Surrogate recovery goals for Tripropyltin were established in the project Sampling and Analysis Plan at 60 to 120 percent for sediment. Based on conversations with the laboratory an additional surrogate, Triphenyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits
Tripropyltin	20 - 195%
Triphenyltin	20 - 172%

Surrogate compound percent recoveries exceeded the QC limits for the following samples

Sample	Surrogate	% Recovery
K980902-MB	Tripropyltin	52
98354086	Triphenyltin	50

As only one surrogate in the samples was outside the QC limits, no qualifiers were assigned based on surrogate results.

7 Laboratory Control Sample (LCS)

LCS recovery goals for Butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment. Based on conversations with the laboratory, historical control chart limits of 20 to 164 percent for sediment were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the following exceptions.

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QA Review Batch K9805842 (Organotin)

Site. Duwamish River

Page 5

LCS	Analyte	% Recovery	QC Limit	Associated Samples
K980902-LCS	Tetrabutyltin	12	60-130 (P) 20-164 (L)	98354060 98354066 98354067 98354083 98354085 98354086
K980902-LCS	Dibutyltin	40	60-130 (P) 20-164 (L)	98354060 98354066 98354067 98354083 98354085 98354086
K980902-LCS	n-Butyltin	14	60-130 (P) 20-164 (L)	98354060 98354066 98354067 98354083 98354085 98354086
K980909-LCS	n-Butyltin	34	60-130 (P) 20-164 (L)	98354070 98354072 98354074
K980909-DLCS	n-Butyltin	24	60-130 (P) 20-164 (L)	98354070 98354072 98354074

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits

8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for sediment

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QA Review Batch K9805842 (Organotin)

Site Duwamish River

Page 6

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
n-Butyltin	10 - 120%

MS/MSD sample percent recoveries met QC guidelines with the exception of the MS and MSD recoveries for Tributyltin in samples 98354085MS and 98354085DMS. Tributyltin recoveries were not calculated due to high analyte concentrations in the sample

Relative percent differences (RPD) between the MS and MSD percent recoveries met QC guidelines for all compounds. No action was based solely on MS/MSD data.

9 Field Duplicate Analysis

No field duplicates were associated with this SDG

10. Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete. A duplicate analysis was performed on sample 98354083. All RPDs were less than 35 percent. The case narrative indicated that the MS and DMS recoveries of Tributyltin in sample 98354085 was not calculated as the analyte concentrations in the sample were significantly higher than the added spike concentration. The case narrative also noted that the LCS recovery of Tetrabutyltin and n-Butyltin were outside the QC limits because of suspected extraction difficulties. As the MS and DMS recoveries were within the QC limits, no further action was taken. No other problems were noted in the case narrative.

11. Laboratory Contact

No laboratory contact was required

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

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QA Review Batch K9805842 (Organotin)

Site: Duwamish River

Page 7

Data Qualifiers

- U - The compound was analyzed for, but was not detected
- UJ - The compound was analyzed for, but was not detected The associated quantitation limit is an estimate because quality control criteria were not met.
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.
- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- N - Presumptive evidence of presence of material (tentative identification).
- I - *Elevated reporting limit due to matrix interference.*

Analytical Report

Service Request: K9805842
Date Collected: 8/26/98
Date Received: 8/27/98

Units ug/Kg (ppb)
Basis Dry

Analyte	Prep	Analysis	MRL	Dilution	Date	Date	Result	Result
	Method	Method		Factor	Extracted	Analyzed		Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	ND	WJ
Tri-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	11	J
Di-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	8	J
n-Butyltin	Method	Butyltins	60	5	9/2/98	9/14/98	ND	WJ B'

Approved By [Signature] Date 10/14/21
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Analytical Report

Service Request: K9805842
Date Collected: 8/27/98
Date Received: 8/28/98

Units	ug/Kg (ppb)
Basis	Dry

D The MRL is elevated because of matrix interferences and because the sample required diluting

Page No
00085

Analytical Report

Service Request: K9805842
Date Collected: 8/27/98
Date Received: 8/28/98

Units	ug/Kg (ppb)
Basis	Dry

00086

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Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805842
Date Collected: 8/27/98
Date Received: 8/28/98

Butyltins

Sample Name	98354070	Units	ug/Kg (ppb)
Lab Code	K9805842-016	Basis	Dry
Test Notes	D		

Analyte	Prep	Analysis	MRL	Dilution	Date	Date	Result	Result Notes
	Method	Method		Factor	Extracted	Analyzed		
Tetra-n-butyltin	Method	Butyltins	5	5	9/9/98	9/16/98	ND	
Tri-n-butyltin	Method	Butyltins	5	5	9/9/98	9/16/98	27	
Di-n-butyltin	Method	Butyltins	5	5	9/9/98	9/16/98	20	
n-Butyltin	Method	Butyltins	5	5	9/9/98	9/16/98	8	J

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By _____ Date 10/19/20

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Page No
00087

Analytical Report

Service Request: K9805842
Date Collected: 8/27/98
Date Received: 8/28/98

Butyltins

Units	ug/Kg (ppb)
Basis	Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	ND	u3
Tri-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	13	5
Di-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	9	↓
n-Butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	6	↓

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By [Signature] Date 10/14/08

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805842
Date Collected: 8/27/98
Date Received: 8/28/98

Butyltins

Sample Name	98354083	Units	ug/Kg (ppb)
Lab Code	K9805842-006	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	ND	WJ
Tri-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	26	J
Di-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	10	J
n-Butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	ND	WJ

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By (4) Date 10/1/98

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10/1/98

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Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805842
Date Collected: 8/27/98
Date Received: 8/28/98

Butyltins

Sample Name	98354085
Lab Code	K9805842-008
Test Notes	D

Units ug/Kg (ppb)
Basis Dry

Analyte	Prep	Analysis	MRL	Dilution	Date	Date	Result	Result Notes
	Method	Method		Factor	Extracted	Analyzed		
Tetra-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	ND	WJ
Tri-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	25	J
Di-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	7	J
n-Butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	ND	WJ

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By

Date 10/17/05

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05842SVG VN1 - 8 10/14/98

Page No

00083

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Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment


Service Request: K9805842
Date Collected: 8/27/98
Date Received: 8/28/98

Butyltins

Sample Name	98354086	Units	ug/Kg (ppb)
Lab Code	K9805842-009	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	5	J ↓
Tri-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	68	
Di-n-butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	24	
n-Butyltin	Method	Butyltins	5	5	9/2/98	9/14/98	10	

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By  Date 10/14/98

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